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Construction of a Chemical Sensor/ Instrumentation Package Using Fiber Optic and Miniaturization Technology

(MSFC Center Director's Discretionary Fund Final Report, Project No. 97-12)

R.L. Newton
Marshall Space Flight Center, Marshall Space Flight Center, Alabama

October 1999

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF ACRONYMS/ABBREVIATIONS

CDDF	Center Director's Discretionary Fund
FOG	fiberoptic gyroscope
LIGA	Lithographie, Galvanoformung, Abformung
MEMS	microelectromechanical systems
SiO_2	silica

TECHNICAL MEMORANDUM

CONSTRUCTION OF A CHEMICAL SENSOR/INSTRUMENTATION PACKAGE USING FIBER OPTIC AND MINIATURIZATION TECHNOLOGY

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1. INTRODUCTION

1.1 Research Motivation

Since the beginning of space flight, lowering the cost of access to space has been a priority. Over the past several years, NASA has refocused its efforts on providing low-cost access to space. The stated goal of reducing the cost of a launch by a factor of 100 by Administrator Golden[1] necessitates the need to employ advanced materials and technologies into launch vehicles. The use of composite materials and more efficient engines allows significant reductions in weight while retaining required strength and thrust. Whether used for instrumentation related to payloads or for vehicle health monitoring, fiber optic and microsystem technology may provide additional savings in weight and volume. The purpose of this research is to construct and evaluate a sensor instrument package using the technologies mentioned above.

1.2 Fiber Optic Technology

The fiber optic industry has experienced tremendous growth in the last several years. This growth has been driven in large part by the communications industry. In 1997 over $9 billion were spent within the United States on fiber optic equipment.[2] To date, 12 billion km of fiber optic cable have been installed in America.[3] This has resulted in making optical fiber a low-cost, easily obtained product.

Optical fibers for light transmission serve as waveguides for the light signals. The retention of light within the optical fiber is made possible by having the light pass through the central core glass which has a higher refractive index than the outer clad glass. It is by monitoring this refractive index change that the fiber can itself be used as a "sensing" device and not only to carry voice or data transmissions. The vast majority of optical fiber is made of silica (SiO_2); however, fibers made of plastic are now commercially available. Optical sensors are capable of measuring a variety of properties,[4] many of which are listed below:

- Temperature
- Pressure
- Flow
- Liquid level
- Electric fields
- Magnetic fields
- Vibration
- Acceleration
- Radiation
- Strain
- Displacement (position)
- Force
- Rotation
- Chemical species
- pH
- Velocity
- Humidity
- Acoustic fields

Many companies have shown particular interest in fiber optic gyroscopes (FOG's). The all solid state nature of the FOG gives it several advantages over their mechanical counterparts. Figure 1 shows the basic components of a FOG. These include low cost, long shelf life, rapid startup, small size, low weight, and rugged construction. FOG's exploit the Sagnac effect, in which two beams traveling in opposite directions around a circle will arrive back at a moving reference point with a phase difference due to rotation.[5]

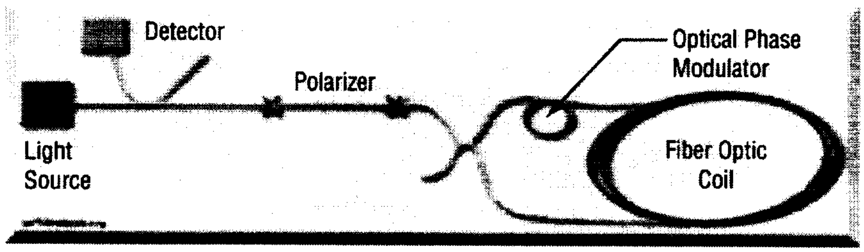

Figure 1. Fiber optic gyroscope.[6]

Fiber optic sensors are currently available for environmental and biological monitoring. Over the past several years fiber optic sensors have been fabricated to measure a range of compounds such as ammonia, oxygen, methane, lead, and many hydrocarbons.[7] Many of these sensors operate by the application of a coating or cladding material to the outside of a bare optical fiber. This cladding material contains molecules that will chemically react with specific target compounds. The interaction of the light with the cladding material when target compounds are present will be of a different wavelength than light interacting with unreacted cladding material. Figure 2 illustrates this specifically for a biosensor,[8] but the principle is essentially the same as that used in a variety of environmental and biosensor applications. This appears to be a very promising technology for the detection of agents that might be used in chemical and biological warfare. One day soldiers will perhaps wear clothing that have these fiber optic sensors woven directly into them and will know instantaneously whether or not they are being exposed to harmful agents.

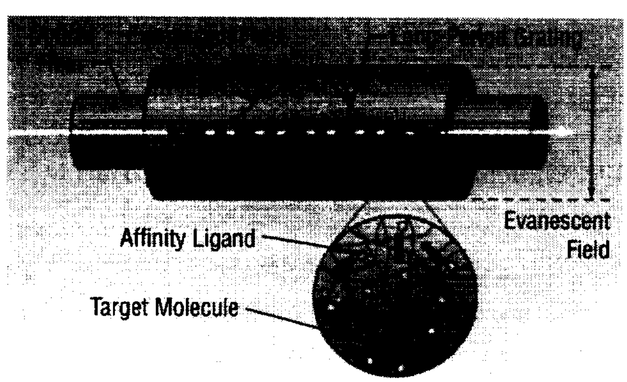

Figure 2. Schematic of cladding applied to optical fiber.

A real strength of fiber optic sensors is that they can act as distributed sensors that can detect changes over the entire length of the fiber, which may be meters or kilometers in length. This technique is being demonstrated by the development of a distributed dosimeter network for detecting hydrazine vapor at rocket launch sites.[9] The effectiveness of this configuration has been demonstrated by using fibers as long as 1 km for sensor applications.

Another promising application of fiber optic sensors is in the area of strain sensors. Operating in this mode, the fibers themselves are monitored for changes in length or optical path of the light beam. This change can be calibrated to movement. This technology is already being applied to the monitoring of civil engineering construction such as bridges.[10] Langley Research Center designed fiber optic sensors to measure strain, temperature, and hydrogen on the X–33 and shuttle fuel tanks. These sensors must operate within an environmental temperature range of –252 to 121 °C and withstand launch and reentry.[11]

1.3 Micromanufacturing/Microsystems Technology

Worldwide, micromanufacturing technology is being employed in automotive, communications, medical, and environmental markets. The global market is estimated at between \$6 and \$14 billion for 1998 and, according to one estimate, is projected upward of \$38 billion by 2002. Much of the current market is focused on accelerometers used in automobile air bag systems and inkjet printer head technology. There are more than 600 companies, universities, and research organizations worldwide currently engaged in this technology.[12] This technology shows particular promise in space-related applications where size, cost, and weight are critical issues in hardware design.

Micromanufacturing, in the narrow sense, comprises the use of a set of manufacturing tools based on batch thin-film fabrication techniques commonly used in the electronics industry. In the broader sense, micromanufacturing describes one of many precision engineering disciplines which take advantage of serial direct write technologies, as well as of more traditional precision machining methods. These tools are used for creating small three-dimensional structures with dimensions ranging from subcentimeters to submicrometers, involving sensors, actuators, or other microcomponents and microsystems.[13] Another term that is often used with respect to micromanufacturing and related fields is microelectromechanical systems (MEMS).

Figure 3 illustrates the scaling of microsystems to other technologies and compares this with the size of items familiar to us. Many of the features and components of MEMS devices are much smaller than a grain of sand. For example, in figure 4 the components of this device are on the order of hundreds of microns. Overall, MEMS is considered an enabling technology, one that will allow for new applications for existing instrumentation and sensing techniques.

A very interesting aspect of MEMS technology is being applied to the area of micropropulsion. One particular research in this area has focused on developing thrusters with impulses of 10^{-4} to 10^{-6} N-sec for high-accuracy station-keeping and attitude control of microspacecraft (e.g. <1 kg).[14] This technology is still in its infancy. Many issues related to the basic properties of materials must be examined on the microscale before MEMS propulsion, or for that matter, many other MEMS devices find widespread use other than the specific applications already mentioned.

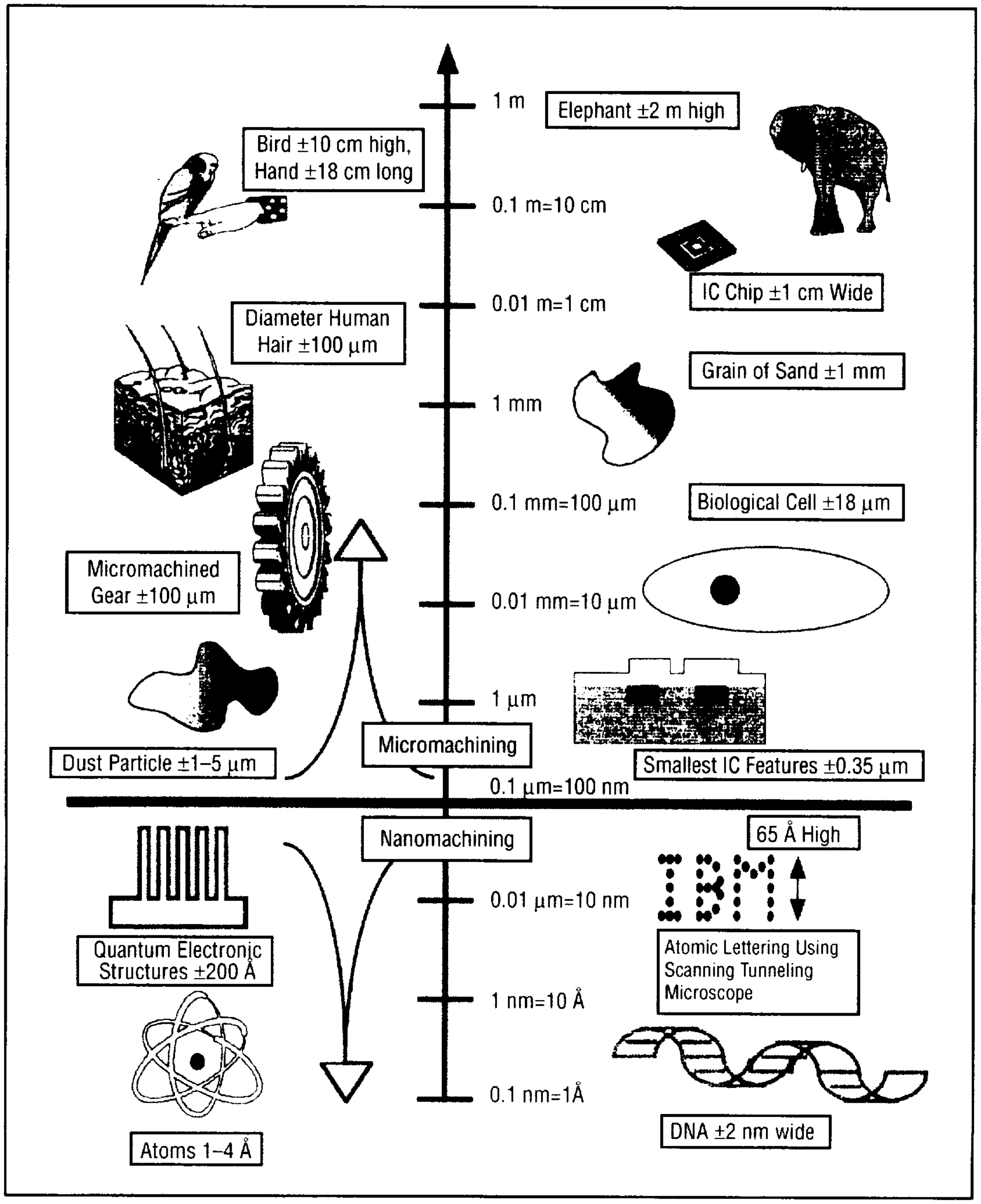

Figure 3. A comparison of the nano, micro, and macro "world".[15]

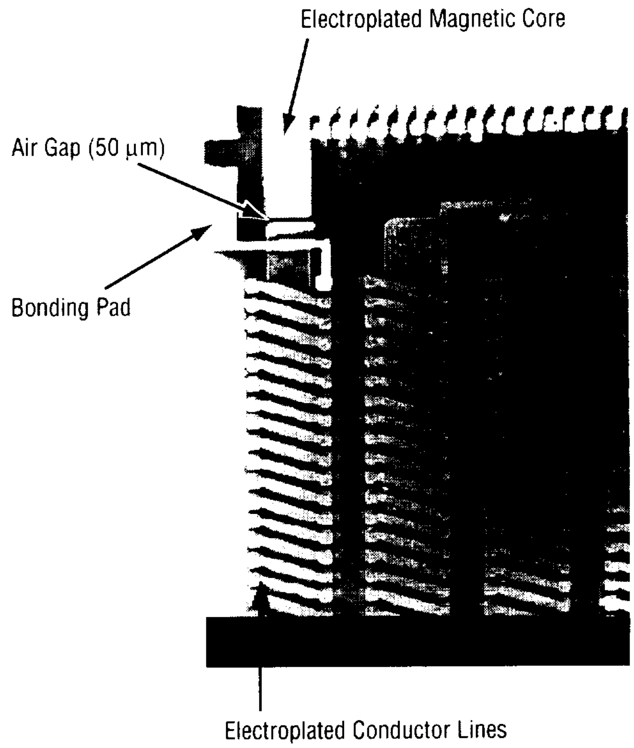

Figure 4. SEM of a top view of the multiturned micromachined inductor.[16]

Spectrometers such as the one used in this investigation have been one of the first analytical tools miniaturized using MEMS technology, due in part to the ability to fabricate microdiffraction gratings. Spectrometers measure the transmission of light through a sample, and according to Beer's law, the absorbance of a solute in a solution is a function of its concentration at a particular wavelength. Thus, absorbance measurements can be used to determine the concentration of solutions.[17]

2. EXPERIMENTAL APPROACH

2.1 Spectrum Selection

The goal of this research was to combine fiber optic sensor technology with miniaturization technology to yield a lightweight, low-cost sensor package. An additional goal of the research was to incorporate wireless data transmission from the sensor package to a computer located some distance away (e.g., 100 ft). It was also decided that the research would initially focus on collecting measurements of species located in the visible region (see fig. 5) of the electromagnetic spectrum due to the availability of miniaturized spectrophotometers manufactured for sensing in the ultraviolet-visible range. Iodine is used in the Space Station water recovery system for disinfectant purposes and has a maximum absorption at 462 nm. Therefore, initial tests were focused on the remote, aqueous detection of this chemical.

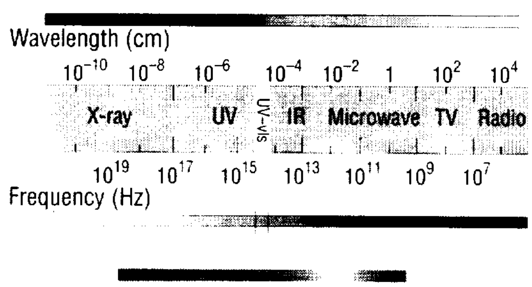

Figure 5. Electromagnetic spectrum investigated in this experiment.[18]

2.2 Spectrometer Selection

The spectrometer selected for the iodine studies is manufactured by microParts[19] and is supplied with a 1-m fiber optic pigtail permanently attached to the spectrometer. Specifications of the spectrometer are given in the appendix. Microsystem technology is employed in this spectrometer via the self-focusing reflection grating, as indicated in figure 6. This particular grating was manufactured using the Lithographie, Galvanoformung, Abformung (LIGA) method.

The light entering the spectrometer via the optical fiber is separated on the curved reflection grating located at the far end of the enclosure. The individual wavelengths of light are then directed back to a diode array which converts the light intensity into an electrical response via a photodiode that is then transmitted to the data system. A diagram of one photodiode that would be coupled with many others to form an array is shown is figure 7. Photodiodes consist of a layer of silicon doped with atoms carrying extra valence electrons (p-type semiconductors) on top of a layer doped with atoms carrying one valence electron less than silicon (n-type) semiconductors.[20]

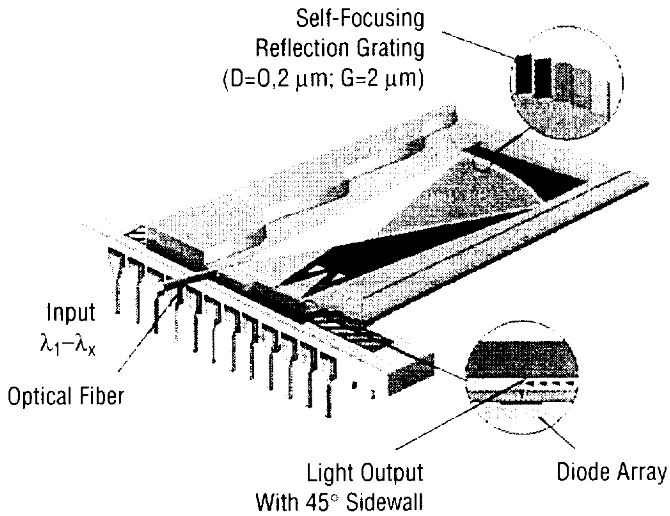

Figure 6. Exploded view of spectrometer diffraction grating.

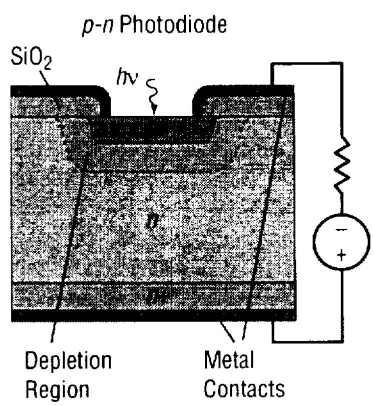

Figure 7. Photodiode cross section.

Since a fiber optic probe was already attached to the spectrometer, the experiments were performed using the spectrometer as a "passive" device. That is, the spectrometer only responded to the amount and wavelength of light that was introduced into the fiber tip. No coating or cladding was applied to the fiber. The test setup for the aqueous iodine solution experiment is given in figures 8 and 9.

The input signal from the fiber optic cable is processed by the microspectrometer and converted to an electrical signal that is relayed via the wireless modem to another wireless modem connected to a PC in a laboratory ≈30 m away. Care must be taken to ensure that no dirt or liquid enters the fiber tip. Also, the fiber will break if bent in too tight of a radius. On this particular model, the fiber is permanently mated to the spectrometer so a broken fiber renders the complete spectrometer useless. The spectrometer measures 13 mm × 35 mm × 2.5 mm.

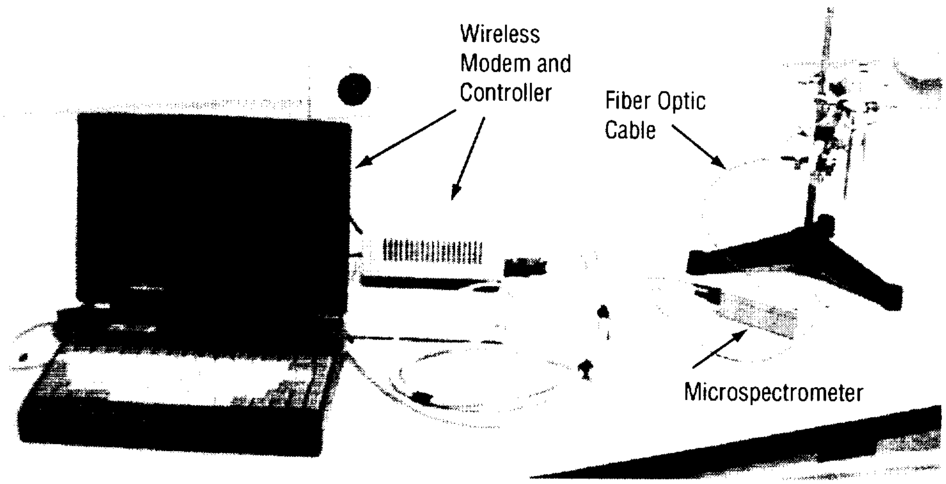

Figure 8. Test apparatus for the sampling of iodine.

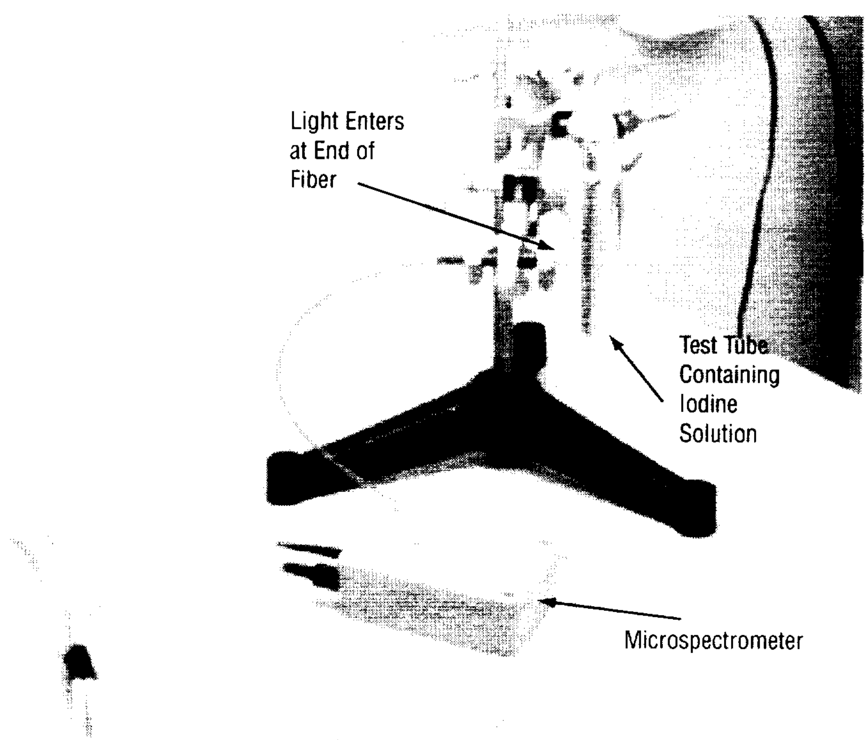

Figure 9. Closeup view of test configuration for iodine analysis.

3. DISCUSSION OF RESULTS

3.1 Experimental Data

In order to test the sensitivity and performance of the spectrometer a set of iodine solutions were prepared at various concentration levels. Additionally, distilled water was used as a "blank" in which no iodine was present. As previously mentioned, iodine has a maximum absorption at 462 nm. Figure 10 demonstrates the spectrum for the blank and figures 11–13 show the spectrum of three concentrations of iodine.

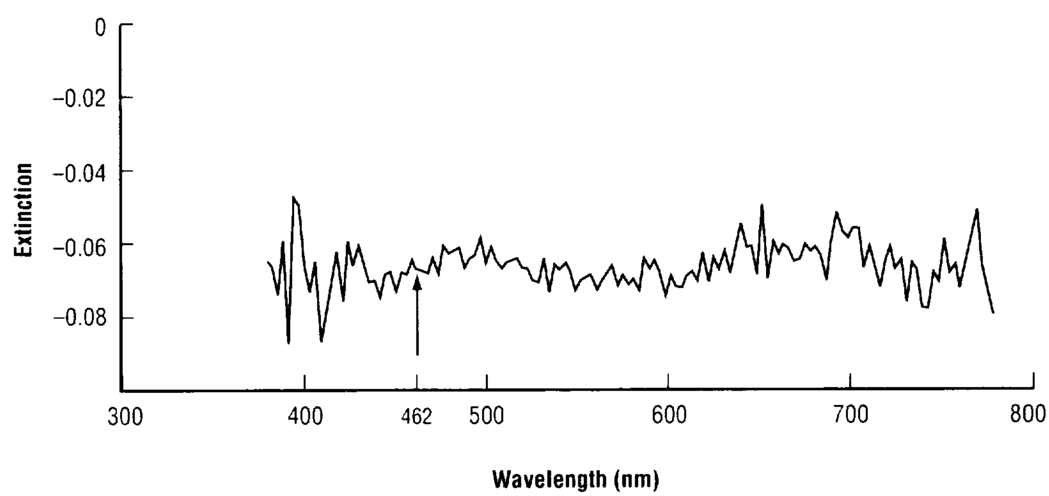

Figure 10. Raw data of spectrum distilled water "blank" used in iodine investigation.

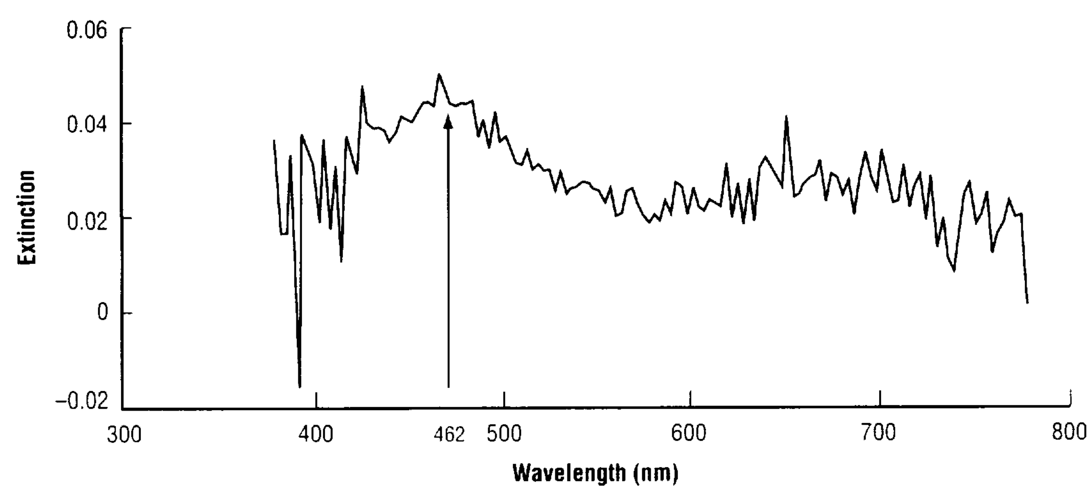

Figure 11. Three parts per million iodine in distilled water.

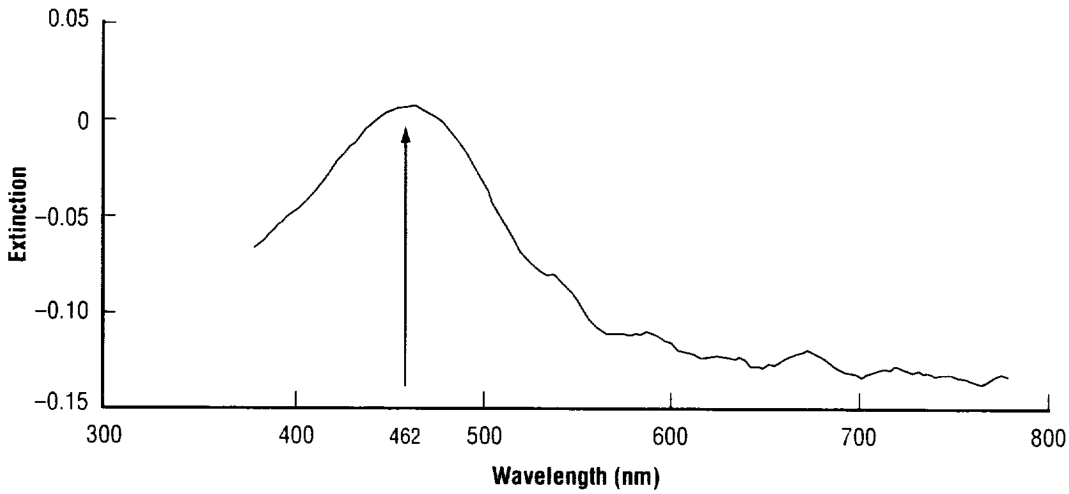

Figure 12. Thirty parts per million iodine in distilled water.

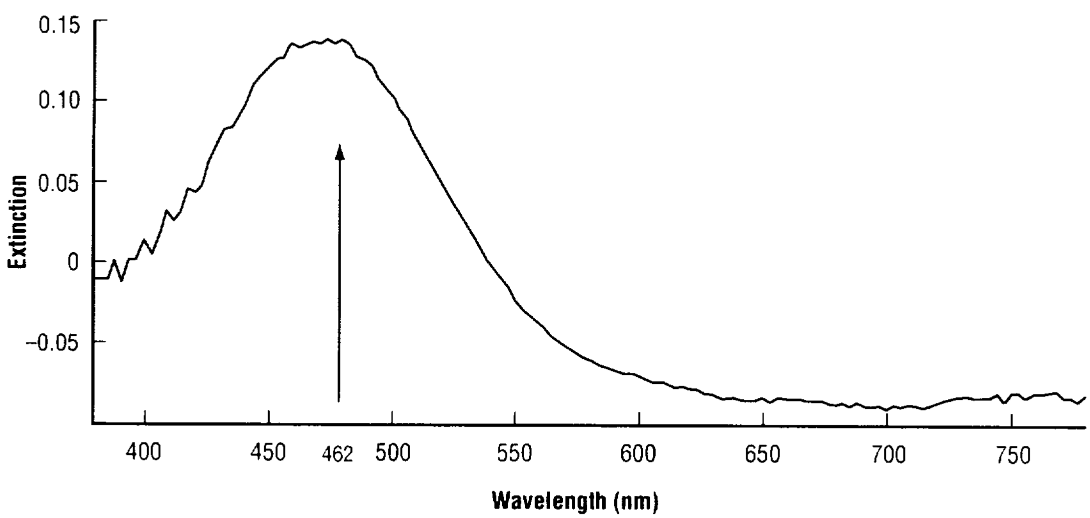

Figure 13. Three hundred parts per million iodine in distilled water.

3.2 Data Analysis and Discussion

The spectrometer exhibited somewhat good sensitivity all the way down to 3 ppm. However, as figure 14 demonstrates, the response does not scale with linear changes in iodine concentration, possibly because of the need for more input signal into the spectrometer at higher dilutions of the iodine mixtures. The use of distilled water should have eliminated any potential for interference from other species.

The wireless data transfer via the radio modem worked well. The range on these particular units is on the order of a few hundred meters, but with modification, wireless data can be transmitted several kilometers, even with low-cost units. Also, in many terrestrial applications of this sensor technology, data could easily be transmitted using the internet.

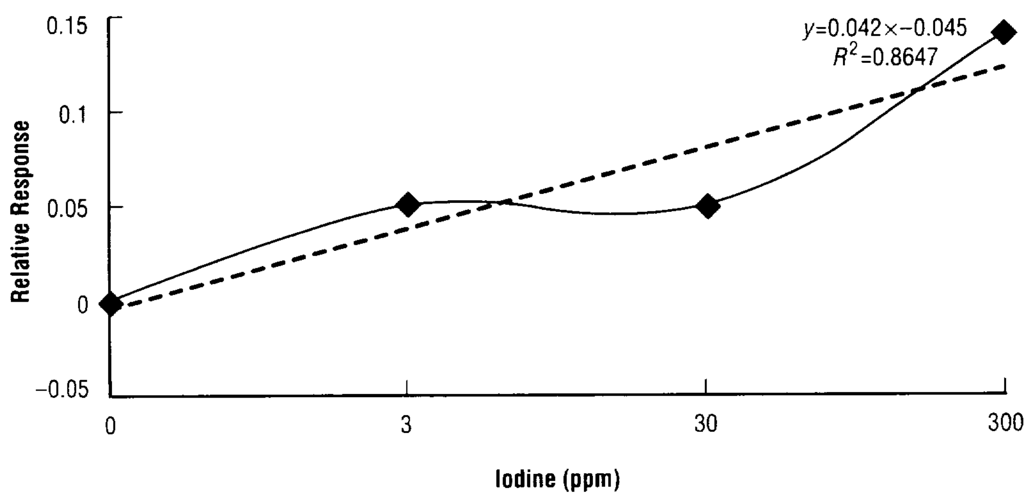

Figure 14. "Raw" data calibration curve for iodine.

4. CONCLUSIONS AND RECOMMENDATIONS

This Center Director's Discretionary Fund (CDDF) was designed as a 2-yr investigation into the construction and testing of an instrument assembly based on fiber optic and miniaturization technology. However, during the first year of this CDDF project, the principle investigator was granted a full-time study award from Marshall Space Flight Center management. In order for this effort to be completed in a timely manner and since the basic objectives of the research were demonstrated, the investigation was only carried through year one.

Fiber optic sensing is a maturing technology. This research examined and demonstrated that the technology is viable for the detection of chemical compounds of interest. Commercially procured sensors are currently available from a wide selection of vendors. The feasibility of using miniaturization technology was also demonstrated. Even on very simplistic experiments, the instrumentation was able to resolve low concentrations (few ppm) of iodine in an aqueous solution.

By the integration of fiber optic sensors and miniaturization technology, size and weight reductions were realized in the construction of an instrument package. Due to the development of this technology by industry and academia, NASA should see positive benefits from future utilization of this technology in its programs.

APPENDIX—Microspectrometer Technical Specifications

Component

Material:	poly(methylmethocrylate)
Dimension:	13.5 nm × 35 mm
Spectral range:	370–850 nm
Blaze wavelength:	560 nm
Grating constant:	1.7 µm
Transmission:	max. 15 percent
Spectral distance:	0.2 nm/µm
Resolution:	7 nm (12 nm) with standard diode array
Order:	first

Fiber

Step index:	50/125 µm or 105/125 µm
Numerical aperture:	0.22
Pigtail length:	1 m
Connector:	Optional

Photodiode Array

Hamamatsu CMOS series or others

Applications

Color measurement

Environmental analysis

Chemical analysis

Medical analysis

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13. ABSTRACT *(Maximum 200 words)*

The objective of this research was to construct a chemical sensor/instrumentation package that was smaller in weight and volume than conventional instrumentation. This reduction in weight and volume is needed to assist in further reducing the cost of launching payloads into space. To accomplish this, fiber optic sensors, miniaturized spectrometers, and wireless modems were employed. The system was evaluated using iodine as a calibration analyte.

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